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EFFECT OF ZINC ON THE SULPHIDATION REACTION
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10/ R. S. Hollingshead
June 1980

Approved by J. R. BROWN

A/Director / Technology Division

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ABSTRACT

One of the major factors controlling the lifetime of marine gas turbines is the rapid degradation of the turbine blades by a process known as sulphidation. In this form of accelerated corrosion attack, the salt ingested with the intake air combines with the sulphur from the fuel and reacts with the blade alloy at high temperature. However, metallurgical examination of turbine blades from a P&W FT-12 gas turbine showed that no degradation of the blades had occurred after 3500 hours of operation in a DDH 280 class destroyer. Similarly, blades from the Solar Saturn gas turbines were sulphidation free after as many as 11,200 hours of operation although degradation due to other causes was observed. The blade surfaces were partially covered by a white deposit identified as $ZnSO_4$. The source of the Zn is the zinc pigmented protective coating in the fuel tanks. Sulphidation of turbine blades was induced in the laboratory where it was found that in the majority of the tests the blades were completely oxidized after 16 - 90 hrs. However, with the addition of 2.5 or 5 percent $ZnSO_4$ to the 90/10 $Na_2SO_4/NaCl$ salt mixture, sulphidation was prevented. These results, along with the observation that sulphidation has not occurred in service, suggest that the presence of Zn in the fuel may have prevented the sulphidation process.

SOMMAIRE

L'un des principaux facteurs qui régit la durée des turbines à gaz montées dans les navires est la détérioration rapide des aubes de turbine selon un processus connu sous le nom de sulfuration. Dans cette forme de corrosion accélérée le sel introduit dans le moteur avec l'air aspiré se combine avec le soufre provenant du carburant et, à des températures élevées, entre en réaction avec l'alliage ferreux dont sont faites les aubes. Cependant, l'examen métallurgique des aubes d'une turbine à gaz P&W FT-12 montée dans un destroyer de la classe DDH 280 a montré qu'il n'y a pas eu détérioration des aubes après 3 500 heures d'utilisation. De même, les aubes des turbines à gaz Solar Saturn n'avaient pas subi les effets de la sulfuration après quelque 11 200 heures de fonctionnement bien qu'on ait constaté qu'il y avait eu détérioration due à d'autres causes. La surface des aubes était partiellement recouverte d'un dépôt blanc; on a découvert qu'il s'agissait du ZnSO_4 . Le Zn provenait du revêtement de protection contenant du zinc qui avait été appliqué aux parois intérieures des réservoirs à carburant. On a provoqué, en laboratoire, la sulfuration d'aubes de turbine et on a découvert que dans la majorité des épreuves, les aubes étaient totalement oxydées après une période de fonctionnement variant de 16 à 90 heures. Cependant lorsqu'on a ajouté 2.5 ou 5 pour cent de ZnSO_4 au mélange de sel $\text{Na}_2\text{SO}_4/\text{NaCl}$ (proportion de 90/10), on a éliminé la sulfuration. Ces résultats, ainsi que le fait que l'on a constaté qu'il n'y a pas eu sulfuration lors du fonctionnement, nous incitent à conclure qu'il se peut que la présence du Zn dans le carburant ait empêché la sulfuration des aubes.

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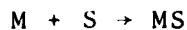
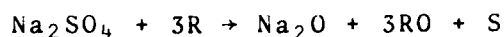
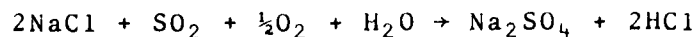
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INTRODUCTION

Early in 1973 the Canadian Forces brought into service the DDH 280 class destroyers. These ships represent a departure from previous Canadian Forces naval vessels in that they are fully gas turbine powered. For propulsion they utilize marinized versions of Pratt and Whitney engines originally designed for aircraft use: Two FT-4's for main propulsion and two FT-12's for cruise. Auxiliary power is provided by three Solar "Saturn" industrial gas turbines. Although these were among the first fully gas turbine powered naval ships in the world, other navies including the British and American had already accumulated considerable experience with gas turbines at sea¹.

Gas turbines ingest air from the atmosphere, compress it, add fuel and burn the mixture. The resulting hot oxidizing gases induce turbine blade temperatures of 650°C to 1000°C. The blade material must therefore have good oxidation resistance as well as good mechanical strength properties to withstand thermal fatigue, creep and stress rupture. Although the design life of gas turbines usually is based on the creep properties of the high pressure rotor materials, the life limiting factor in marine installations generally has been the ability of the material to resist the accelerated oxidation reaction: Sulphidation.

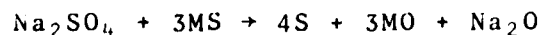
This form of corrosion especially affects the turbine blades and the inlet nozzle guide vanes which direct the hot gases into the turbine. It occurs in the presence of sulphur which is found in the fuel and of sodium which is ingested as sodium chloride with the intake air. Hardt et al² concluded that the salt takes part in the sulphidation reaction by providing Na for the formation of Na₂SO₄ as follows:



M = metal

R = unspecified reducing agent

The autocatalytic conversion of the metal sulfide to oxide follows:



These reactions occur between 750° and 1000°C. The upper and lower limits vary according to the amount and nature of the contaminant and blade material.

More recently Conde³ has pointed out that NaCl is liquid over the temperature range in which the attack takes place and that liquid salt will disrupt the otherwise protective oxide layer. Seyboldt⁴ also found that sulphur can induce oxidation of the blade material. He theorized that the formation of sulphides in the matrix of the blades leads to migration into the grain boundaries of those elements which inhibit oxidation. The grains, impoverished by this migration, then suffer rapid oxidation. There is growing evidence⁵ that further oxidation of some alloying element oxides which would otherwise assist the formation of a protective oxide layer occurs in the presence of Na₂O: $4\text{Na}_2\text{O} + 2\text{Cr}_2\text{O}_3 + 3\text{O}_2 \rightarrow 4\text{Na}_2\text{CrO}_4$.

The rate at which sulphidation occurs can be diminished by reducing hot section temperature; that is, by operating at low power and minimizing periods of high power operation, and by minimizing the quantity of salt reaching the engines. For these reasons the DDH 280 propulsion engines are generally operated at low power and the air intakes are fitted with demisters consisting of a series of angled polypropylene knit mesh filter pads.

In order to evaluate the effectiveness of these safeguards, several specimen hot section parts such as nozzle guide vanes and turbine blades were examined after varying lengths of service. Although some of these showed deterioration, none have showed any sign of sulphidation or other form of degradation attributable to operation in the marine environment. Figure 1 shows a nozzle guide vane and Figure 2, a first stage turbine blade from an FT-12 engine after 3038 hours of operation.

These parts, made of diffusion coated AMS 5385 alloy (Table I) exhibited only a slight erosion of the coating, probably due to carbon particles. The white deposit was identified by X-ray diffraction as zinc sulphate. Similarly the Solar "Saturn" turbine blade shown in Figure 3, which is made of S-816 alloy (Table I), suffered erosion damage during 11,200 hours of service but no sulphidation. Again the white deposit was identified as zinc sulphate.

The presence of zinc sulphate is attributable to the use of an inorganic zinc-rich protective coating in the fuel tanks of the DDH 280's. During the early life of these ships

zinc concentrations in the fuel exceeded 100 ppm. However after 1 to 2 years service the concentration had fallen to about 5 ppm. Presently the concentration is approximately 0.5 ppm.

That there was no sign of sulphidation whatever, even though the normal operation of these ships is such as to minimize its incidence, suggested that a constituent of the hot section environment peculiar to the gas turbines of the DDH 280's might be acting to prevent it from occurring. Such a constituent could be the zinc leached by the fuel from the fuel tank coating. As already noted, zinc, combined with sulphur as zinc sulphate, is regularly observed on the surfaces of DDH 280 engine hot section parts. In order to test this hypothesis, a series of laboratory trials was conducted in which portions of hot section parts were subjected to a crucible test used by Schultz et al⁶ to induce sulphidation.

EXPERIMENTAL PROCEDURE

In the crucible test the specimen is partially immersed in a salt mixture in a ceramic crucible (Figure 4) and heated in an oven at 900°C. The salt mixture used by Schultz⁶ to induce sulphidation consisted of 90 weight percent sodium sulphate and 10 weight percent sodium chloride.

In order to see whether the presence of zinc would influence the crucible reaction, salt mixtures were utilized in which either 2.5 or 5 weight percent of sodium sulphate was replaced by zinc sulphate.

Most of the specimens consisted of the mounting portions ("fir tree" sections) of Solar "Saturn" turbine blades. These are made, as is the entire blade, of the super-alloy S-816, the composition of which is given in Table I. In addition, a few sections of Pratt and Whitney FT-12 nozzle guide vanes were also included. These are made of the super-alloy AMS 5385, the composition of which is also provided in Table I, and diffusion coated for added resistance to sulphidation and erosion.

Thirty-eight exposures were carried out. In these exposures, crucibles containing $\text{Na}_2\text{SO}_4/\text{NaCl}$ or $\text{Na}_2\text{SO}_4/\text{NaCl}/\text{ZnSO}_4$ mixtures were placed in the furnace simultaneously and the specimens examined periodically. When the specimens

immersed in the $\text{Na}_2\text{SO}_4/\text{NaCl}$ mixture showed signs of attack or complete oxidation, they were removed from the furnace. The specimens immersed in the mixture containing ZnSO_4 were left for longer periods depending on the availability of the furnace in an effort to achieve sufficient attack for morphological study. In the case of the FT-12 specimens, two crucibles containing specimens immersed in $\text{Na}_2\text{SO}_4/\text{NaCl}$ and two containing $\text{Na}_2\text{SO}_4/\text{NaCl}/\text{ZnSO}_4$ were placed in the furnace and removed together.

The extent of attack for each exposure was determined by measuring the cross-sections of the specimens before and after the exposure (Figure 5).

Those specimens which were not completely oxidized were metallographically mounted, polished and etched. The morphology of the attack and the microstructure were examined using both a metallographic microscope and a scanning electron microscope.

RESULTS

Table II, which shows the results of exposure to a 90/10 mixture of Na_2SO_4 and NaCl , indicates that 12 of 18 samples were completely oxidized in exposure times of 16 to 90 hours (Figure 6). Only two blades showed no deterioration after exposure times of 40 and 70 hours although several others displayed a low level of sulphidation. In marked contrast, the results of Table III, for exposures to mixtures containing either 2.5 or 5.0 percent ZnSO_4 for times from 30 to 112 hours, show nil or little wastage in all 20 cases and the wastage that occurred was ascribed to normal high temperature oxidation.

The study of the morphology of the specimens exposed to zinc sulphate-free salt revealed that sulphur migrates into the matrix via the grain boundaries where it reacts with the chromium of the matrix to form chromium sulphides. These sulphides are formed at or near grain boundary carbides in advance of the matrix/scale interface (Figure 7) and then branch out into the chromium depleted areas of the surrounding matrix. The resulting scale was relatively voluminous, porous and poorly adherent (Figures 8 and 11). On the other hand, the microstructure of those specimens exposed to the salt mixture containing zinc sulphate revealed no sulphides in the matrix (Figure 9) and the scale formed, of which there was very little, was very compact and adherent (Figures 10 and 11) similar to that found on the samples removed from the ship's engine.

DISCUSSION

The choice of materials for the hot sections of the gas turbines was dictated in part by the requirements for sulphidation resistance. However, that these materials are not immune was demonstrated by the crucible test. Although the attack on the specimens exposed to the zinc-free salt mixture varied considerably in extent, it is important to note that it affected 16 of the 18 specimens and that the morphology of the residue after exposure was typical of that described in the literature for sulphidation. On the other hand, none of the 20 specimens exposed to the salt mixture containing zinc sulphate showed any sign of sulphidation and the specimen morphology after exposure closely resembled that of the hot section parts from the DDH 280 gas turbines.

Although the conditions in the DDH 280 propulsion engines are such as to minimize the rate at which sulphidation occurs, the hot section temperatures are in the sulphidation range some of the time and measurements indicate that appreciable quantities of salt reach the engine in spite of the air intake filters. In the case of the Solar "Saturn" engines, temperatures are normally in the sulphidation range and the intake air filters are less efficient than those of the propulsion engines. Thus, it is unlikely that careful handling and operating of the engines can alone account for the total absence of sulphidation. Rather, the absence of sulphidation in the DDH 280 gas turbines points to the influence of a constituent of the hot section environment such as zinc which is particular to these engines.

The evidence for a causal relationship between the presence of zinc in the fuel supplied to DDH 280 gas turbines and the absence of sulphidation in those same engines can be summarized as follows:

1. Sulphidation of DDH 280 hot section parts can occur;
2. DDH 280 engine hot section parts do not suffer sulphidation in the service environment;

3. Sulphidation of these parts can be prevented in the laboratory by the addition of zinc sulphate to a sodium sulphate sodium chloride mixture;
4. Zinc is present in the fuel supplied to the DDH 280 gas turbines;
5. Zinc sulphate is known to deposit on DDH 280 engine hot section parts;
6. What little oxide forms in service corresponds closely, both chemically and physically, to that formed on the laboratory specimens exposed to salt mixtures containing zinc sulphate.

Thus it appears that the presence of zinc in the combustion environment has probably prevented the occurrence of sulphidation in the DDH 280 gas turbines. This being the case, it should be possible to provide for the presence of controlled quantities of zinc in the combustion chamber specifically for the control of sulphidation. As noted earlier, the concentration of zinc in the fuel currently supplied to the DDH 280 engines is now only about 0.5 ppm. Whether or not this level is adequate for preventing sulphidation is a question to be answered by further examination.

CONCLUSIONS

It has been found that, although DDH 280 hot section parts have suffered no sulphidation in service, they are not immune to this form of corrosion which has been induced in the laboratory. However, the presence of zinc, which is also peculiarly present in the DDH 280 fuel supply, prevents sulphidation in the laboratory experiments. Thus it is strongly indicated that sulphidation of marine gas turbine hot section parts can be prevented by the introduction of zinc into the combustion environment.

TABLE I

SPECIMEN COMPOSITIONS

1st Stage Solar Saturn
Turbine Blade S-816

C	.3 - 4 %
Cr	19.0 - 21.0 %
Co	40.0 % min
Nb	3.5 - 4.5 %
Fe	5.0 % max
Mn	1.0 - 2.0 %
Mo	3.5 - 4.5 %
Ni	19.0 - 21.0 %
W	3.5 - 4.5 %

FT-12 Nozzle Guide
Vane AMS 5385

C	.2 - .3 %
Cr	25.0 - 29.0%
Co	67 % min
Fe	3.0 % max
Mn	1.0 % max
Mo	5.0 - 6.0 % max
Ni	1.8 - 3.8 % max

TABLE II

RESULTS OF EXPOSURE TO 90/10 Na₂SO₄/NaCl at 900°C

<u>Exposure Time</u> <u>(Hours)</u>	<u>Wastage (cm)</u>
Solar "Saturn" Turbine Blades	
16	Complete
18	Complete
18	Complete
18	Complete
40	Nil
41	Complete
48	0.036
55.5	Complete
64	0.003
64	Complete
64	Complete
70	Nil
70	Complete
70 3/4	0.170
71	0.005
80	Complete
FT-12 Nozzle Guide Vanes	
30	Complete
30	Complete

TABLE III

RESULTS OF EXPOSURE TO SALT SOLUTIONS CONTAINING
ZINC SULFATE AT 900°C

<u>Percent ZnSO₄ in</u> <u>Solution</u>	<u>Exposure Time</u> <u>(Hours)</u>	<u>Wastage (cm)</u>
Solar "Saturn" Turbine Blades		
2.5	55.5	Nil
2.5	55.5	Nil
2.5	64	0.002
2.5	64	0.008
2.5	64	0.009
2.5	70	Nil
2.5	71	0.006
2.5	71	0.010
2.5	72	0.008
2.5	80	0.029
2.5	96	0.015
2.5	112	0.002
5.0	72	0.014
5.0	72	0.034
5.0	96	Nil
5.0	96	0.018
5.0	112	0.002
5.0	112	0.027
FT-12 Nozzle Guide Vanes		
2.5	30	Nil
2.5	30	Nil

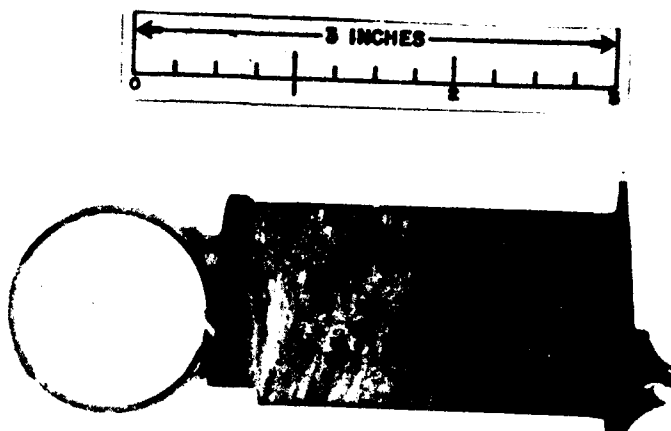


FIGURE 1: FT-12 nozzle guide vane after 3038 running hours.

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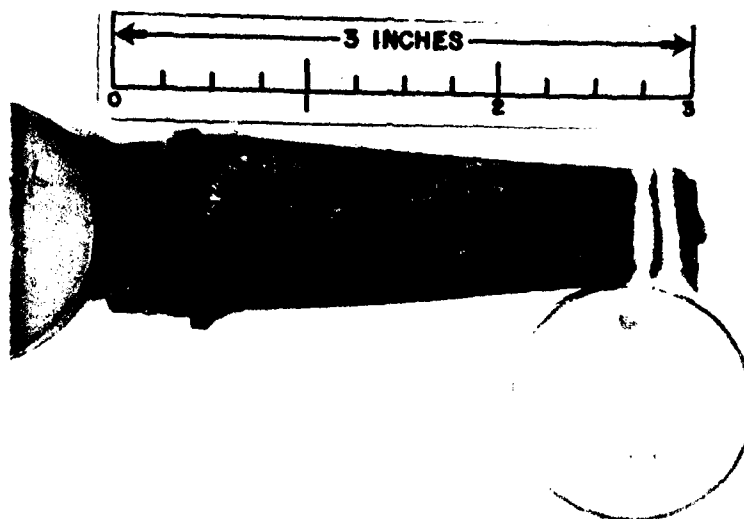


FIGURE 2: FT-12 first stage turbine blade after 3038 running hours. White deposit is ZnSO_4 .

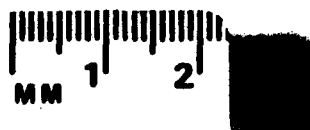


FIGURE 3: Solar "Saturn" 1st stage turbine blade after 11,200 running hours.



← 5 cm →

FIGURE 4: Fir tree of Solar "Saturn" 1st stage turbine blade partially immersed in salt mixture.

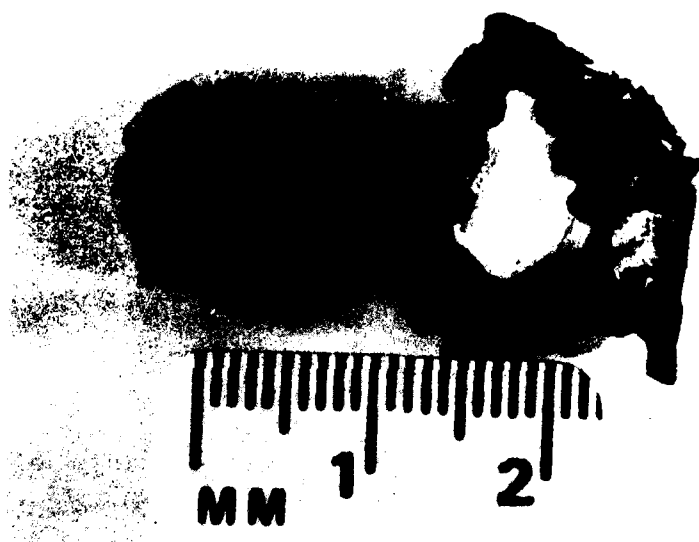


FIGURE 5: Left - Fir tree before exposure
Right - Fir tree after exposure to 90% Na_2SO_4 , 10 %
NaCl for 25 hours.



FIGURE 6: Solar "Saturn" 1st stage turbine blade completely oxidized after 30 hours.

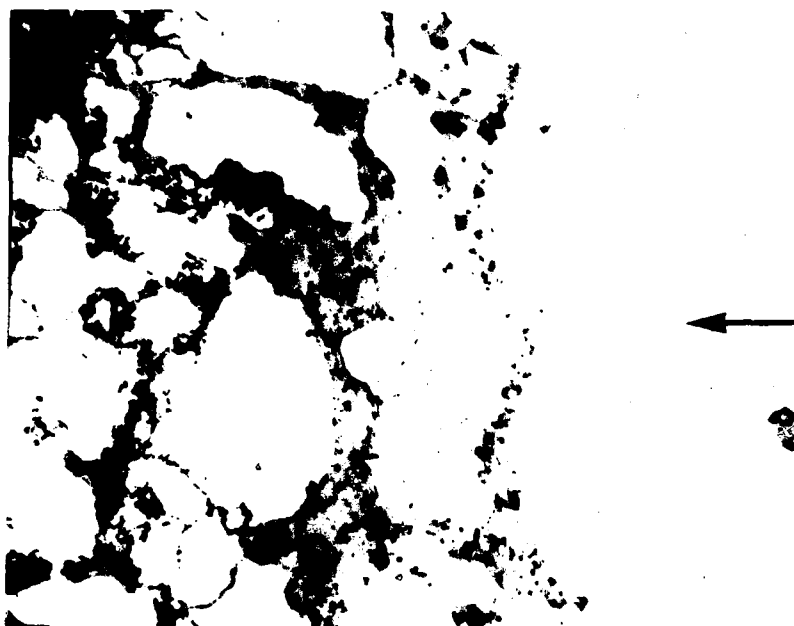


FIGURE 7: The grey areas at the grain boundaries are chromium sulphides. Arrow indicates direction of scale/metal interface.

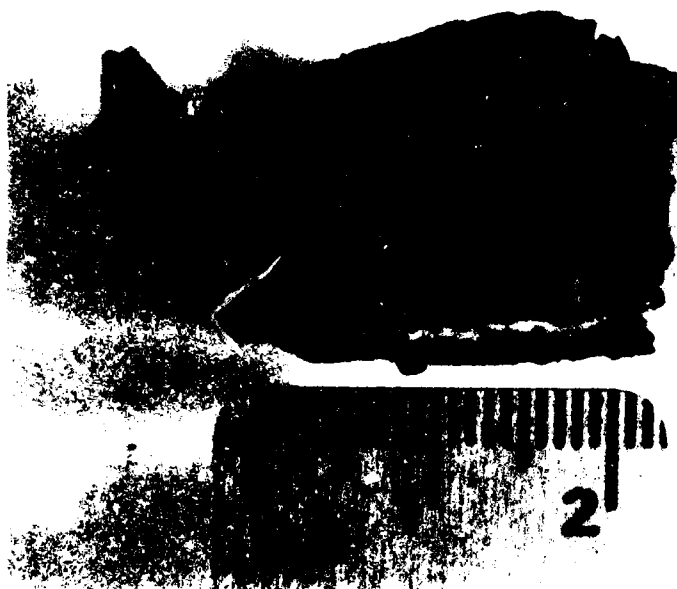


FIGURE 8: Porous non-adherent scale formed in absence of ZnSO_4 .

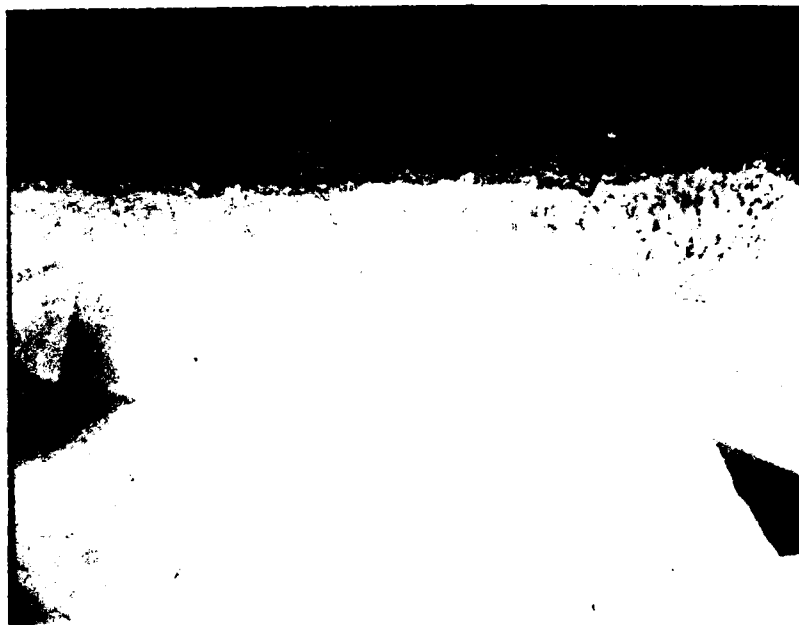


FIGURE 9: Scale/metal interface of blade subjected to salt mixture containing ZnSO_4 . Scale is mainly chromic oxide and zinc oxide.



FIGURE 10: Compact adherent scale formed in presence of ZnSO_4 .

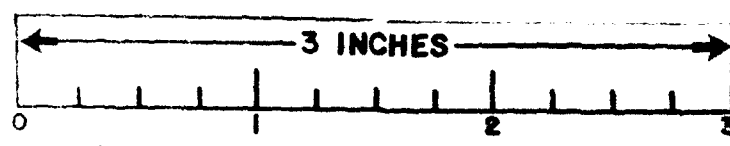


FIGURE 11: Left - Two FT-12 nozzle guide vanes immersed in 90/10 $\text{Na}_2\text{SO}_4/\text{NaCl}$ for 20 hours.

Right - Two FT-12 nozzle guide vanes immersed in 88.5/10/2.5 $\text{Na}_2\text{SO}_4/\text{NaCl}/\text{ZnSO}_4$ for 20 hours.

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<p>One of the major factors controlling the lifetime of marine gas turbines is the rapid degradation of the turbine blades by a process known as sulphidation. In this form of accelerated corrosion attack, the salt ingested with the intake air combines with the sulphur from the fuel and reacts with the blade alloy at high temperature. However, metallurgical examination of turbine blades from a P&W FT-12 gas turbine showed that no degradation of the blades had occurred after 3500 hours of operation in a DDH 280 class destroyer. Similarly, blades from the Solar Saturn gas turbines were sulphidation free after as many as 11,200 hours of operation although degradation due to other causes was observed. The blade surfaces were partially covered by a white deposit identified as $ZnSO_4$. The source of the Zn is the zinc pigmented protective coating in the fuel tanks. Sulphidation of turbine blades was induced in the laboratory where it was found that in the majority of the tests the blades were completely oxidized after 16 - 90 hrs. However, with the addition of 2.5 or 5 percent $ZnSO_4$ to the 90/10 Na_2SO_4/$NaCl$ salt mixture, sulphidation was prevented. These results, along with the observation that sulphidation has not occurred in service, suggest that the presence of Zn in the fuel may have prevented the sulphidation process.</p>		

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